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First Quarterly Progress Report
Contract No. NASw-734

INVESTIGATION OF ADHESION AND COHESION OF METALS IN ULTRAHIGH VACUUM

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FOREWORD

This is the first quarterly report of work performed in the Research Division of National Research Corporation under Contract No. NASw-734 for the National Aeronautics and Space Administration.

The general object of the work is to obtain additional information as to the conditions under which metals and alloys of engineering importance for space applications will adhere to one another with sufficient tenacity to hinder the relative motion or subsequent separation of components of mechanical and electrical devices used in space exploration. Such devices include, bearings, solenoids, valves, slip rings, mating flanges, conical rendevous mating surfaces, etc.

The materials to be studied are copper, copper-beryllium alloy, 1018 steel, 4140 steel, 440C stainless steel, titanium and an electrical contact alloy. The major variable is hardness but ion bombardment cleaning is to be compared with wire brushing.

This work is a continuation of work on the cohesion of copper to copper and of steel to steel performed under NASA Contract No. NASr-48.

ABSTRACT

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Apparatus used in previous work for studies of cohesion (bonding of similar metals) is being modified to permit studies of adhesion (bonding of dissimilar metals).

Indexing devices permitting tests on eight sample pairs for a single vacuum pumpdown operation have been built and are described.

Modification of the spool piece which houses the apparatus was required and is partially complete.

Adhesion studies require a surface cleaning operation not required in the previous cohesion work in which specimens were fractured and rejoined. Two ion guns and a wire-brushing device are under construction for such cleaning. All of the parts for the ion guns have been made and will be assembled, and mounted on the spool piece as soon as parts, which were sent out for a special brazing operation, are received. The ion gun design is discussed herein, and the reasons for using guns rather than simple glow discharge are discussed.

Design of a mechanism for cleaning by wire brushing is complete but it has not yet been assembled or mounted. The possibility of obtaining brushes with tungsten bristles held between twisted stainless steel wires is being investigated and it appears that they may be obtainable from a local vendor.

The sample materials are on hand or on order except for the electrical contact alloy. Its selection awaits further literature survey. Plans for the remainder of the work are presented.

INTRODUCTION

Previous work indicated that hardness was an important variable with respect to cohesion. (This work was described in the First Annual Summary Report - "Investigation of Adhesion and Cohesion of Metals in Ultrahigh Vacuum" - by John L. Ham, dated Sept. 7, 1962, under Contract No. NASr-48, National Research Corporation Project No. 42-1-0121 and Reference No. N62-17772.) Specifically, it was found that copper or mild steel when repeatedly fractured and rejoined in vacuum exhibited less and less cohesion even at ambient pressures believed to be too low to permit significant contamination in the time available. These initially soft metals were work hardened by this treatment and the successive reductions in cohesion are ascribed to this hardening. No cohesion could be measured on initially hard heat treated 52100 steel at room temperature when tested in a similar manner.

Although there are many other important variables such as temperature, time in contact, degree of deformation in compression, sliding, etc., it is considered most important, from a practical standpoint, to first assess the tendency of various commonly used alloys to stick together at room temperature without sliding or severe deformation. Furthermore, it seems most important to determine the behavior of truly clean surfaces before attempting to study the complex variable of degree of contamination. Therefore, the experiments are planned so as to provide clean surfaces by either ion bombardment or wire brushing (whichever proves to give the most adhesion) in vacuum. Exposure to vacuum alone at room temperature can remove only certain physisorbed gases and only mild heating in vacuum can be tolerated when the effect of hardness conferred by work hardening or even by heat treatment is to be evaluated. Within this frame of reference, plans have been made for the necessary modifications to the equipment previously used and tentative procedures were also laid out. These plans are summarized by the seven specific tasks listed below:

Task A - Modify the experimental apparatus used in the performance of the contract NASr-48 by introduction of one or more ion guns for ion bombardment cleaning of surfaces, and introduction of mechanical devices for cleaning under vacuum by filing or brushing.

Task B - Modify the system to permit loading of several pairs of specimens for each pumpdown of the system

Task C - Compare ion bombardment cleaning with abrasion cleaning using cohesion of soft O.F.H.C. copper to soft O.F.H.C. copper and soft 1018 steel to soft 1018 steel as the comparison criterion, since data are available from the previous studies on the cohesion of these metals using the "make-break" rupture technique. After this comparison, one cleaning method shall be selected for subsequent studies on cohesion and adhesion of clean surfaces.

Task D - Measure the adhesion of soft O.F.H.C. copper to each of six other metals, at room temperature, for both the hard condition and soft condition, of the second metal. Tentatively, the other metals will be 1018 steel, 440C steel, 4140 steel, Cu-Be alloy N025, an electrical contact alloy and commercially pure titanium. All surfaces shall be precleaned in vacuum by the method selected under Task C.

<u>Task E</u> - Measure the cohesion of the five metals listed under Task D at room temperatures with the metals in both hard condition and in soft condition. The cohesion of copper and 1018 steel shall be measured in the hard condition. (Data on the latter two metals in soft condition are already available from Task C.)

<u>Task F</u> - Measure the cohesion of all seven metals at room temperature with one half of the pair in hard condition and the other specimen in soft condition.

<u>Task G</u> - Study other variables as time permits. Other variables of importance to adhesion and cohesion are degree of deformation of one or both specimens at the interface, temperature of the specimens, time in contact and degree of contamination of the surfaces.

Task A and B are partially complete. Specimen preparation is under way and the vacuum system to be used, which is the newest and fastest of its type in the NRC Space Vacuum Laboratory, has been checked out. This report deals with the details of these tasks.

MODIFICATION OF APPARATUS

The changes required to permit multiple sample loading and cleaning by ion bombardment were quite extensive. It is necessary to provide indexing so that a given specimen can be oriented first for bombardment, then for testing. By using 45° indexing, eight specimen pairs can be loaded. This modification is illustrated by Figs. 1 and 2 which show schematically the apparatus before and after modification.

Fig. 2 does not convey the true degree of complexity of the modified apparatus. Numerous windows, and electrical and mechanical feedthroughs are required in the wall of the vacuum enclosure. Four bellows mechanical feedthroughs are required for indexing and locking the two wheels and one for wire brushing. Shadowgraph-type specimen observation and measurement during testing requires two windows and a mirror.

The ion guns to be described later are to be mounted in 4 inch-diameter side arms, each with seven electrical and one gas feedthrough. Windows are required for ion beam fluorescent target observation during alignment. A device for monitoring the cleaning operation by measuring the Auger effect (electron emission from the target) may have to be employed.

The apparatus (Fig. 2) will sit on top of the vacuum system which consists of a 14-inch diameter stainless steel bowl with a 10-inch diameter elbow leading from its side to a chevron type liquid nitrogen cooled trap, on top of a 10" diffusion pump with a "cold cap."

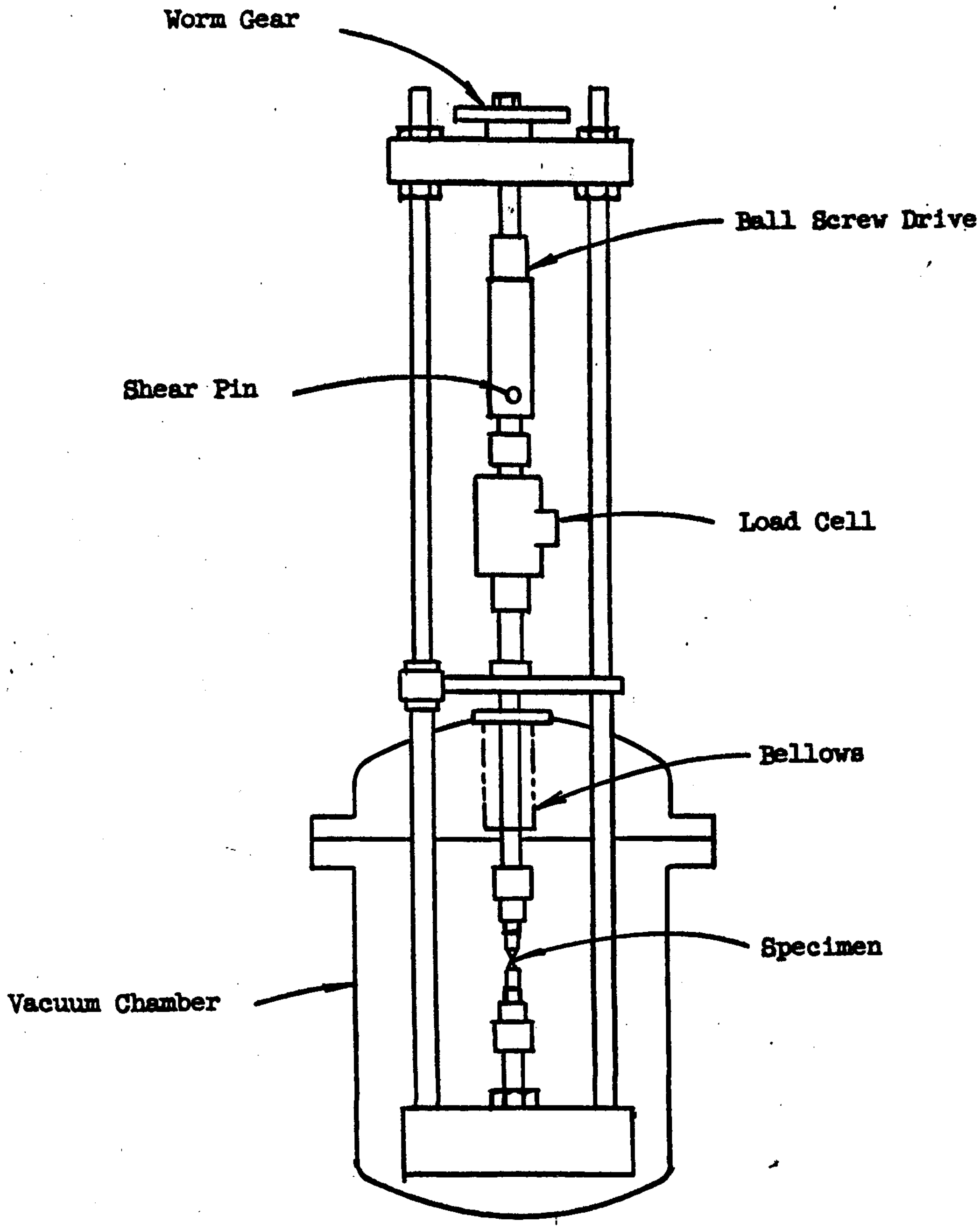


FIGURE 1 - Cohesion Test Apparatus

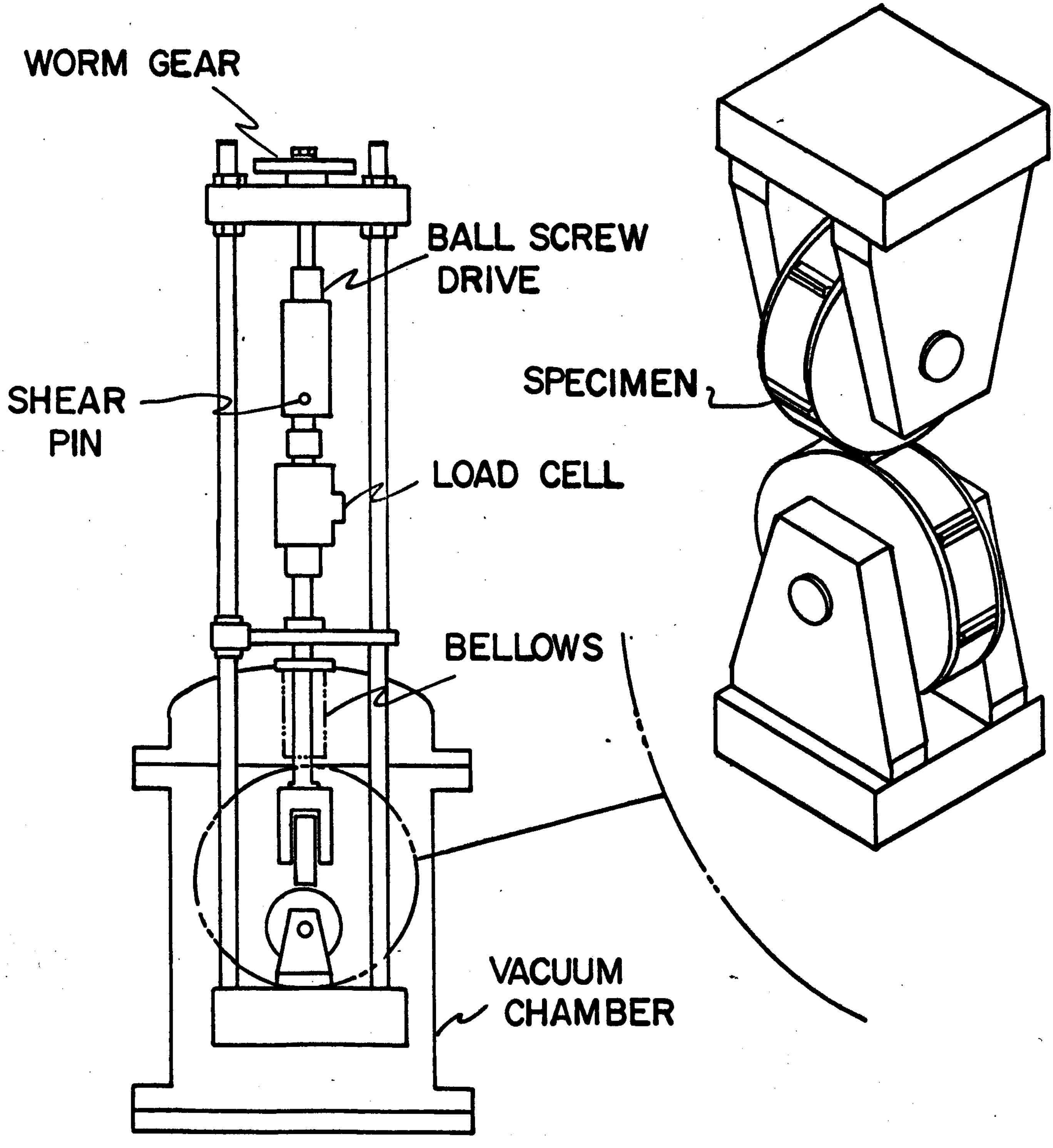


FIGURE 2 - COHESION TEST APPARATUS

The usual UHV type ionization vacuum gauge is used. A pressure of 2 x 10^{-9} torr has been obtained with no bake-out.

CLEANING BY ION BOMBARDMENT

Ion bombardment cleaning is frequently accomplished in small glass systems by simply initiating an electrical discharge between the sample and an adjacent positive electrode with the whole system filled with the gas to be ionized at a pressure of about 10^{-2} to 10^{-1} torr. However, this requires isolation of the system from the diffusion pumps and in the system in use for the cohesion studies valving adds considerable expense. There is also uncertainty as to the actual composition of the gas over extended periods of time, even though the relatively large volume of gas required is initially pure. Finally, a finite time would be required to reduce the system pressure from 10^{-2} torr to the 10^{-8} to 10^{-9} torr range desired for the experimental environment and recontamination of surfaces might occur during this pumpdown period. Therefore, it was decided to use an ion gun which can direct a beam of ions at the specimen surface only thus permitting the system to operate at low pressures even during the cleaning period.

The ion gun can maintain the pressure of 10^{-2} torr within the gun cavity required to maintain the discharge. An ion beam is emitted through an orifice and is directed to the target specimen. Some idea of the pressure which can be maintained in the system can be obtained by comparing the amount of gas typically effusing from the gun with the pumping speed of the system. A gun with an internal pressure of 10^{-2} torr and an orifice of 0.010-inch diameter would effuse approximately 6×10^{-5} torr liters per second of air. If we assume a net pumping speed of 1000 liters per second at the exit from the test system then the system pressure would be 6×10^{-8} torr with the ion gun operating. The advantage of the ion gun over a local discharge to the specimen in a large chamber is that the ratio of the impingement rate of the desired species (Xe^+, for example) to that of undesired species (O_2, O^+, CO, CO^+, CO^{++}, etc.) is larger and more precisely known. Furthermore, the energy and species (Xe^+ or Xe^{++}) of the impinging inert gas ions is more controllable. The number of impinging nonionized inert gas atoms may be greater or less than in a local discharge but this is of little consequence.

It is important to control the energy of the impinging ions since ions even of the inert gases penetrate into the metal lattice if they impinge with too great an energy. This is shown by curves of "sticking coefficient" versus voltage, obtained by measuring the amount of inert gas given off on subsequent heating in vacuum. Apparently, the ions actually form substitutional solid solutions with metals and diffuse out on subsequent heating according to the usual diffusion laws. However, the sticking coefficient curves indicate zero sticking (penetration) below a critical voltage for each type of ion. For example, argon ions penetrate into tungsten above 150 volts but Xe ions require 200 volts.

To remove inert gases from metals requires relatively high temperatures since the activation energies of diffusion appear to be comparable to that for self-diffusion of the metal itself. The apparatus being constructed is not suitable for high temperature specimen outgassing,and since the magnitude of the effect of inert gas ions in solid solution on hardness and strain hardening coefficient is not known it seems advisable to stay below the critical sticking coefficient voltages if possible. Data in the literature indicate that good "cleaning" (ratio of sputtered atoms or ions to impinging ions) is possible below these voltages but there is some question as to how efficiently the ion guns can be made to work at such low voltage. Obviously, the heavier the impinging ion the better, since a larger percentage of the energy is given up near the surface and higher voltage can be used without penetration. Therefore, xenon rather than argon will probably be used. Actually, it is almost impossible to clean metal surfaces in vacuum at low temperature without changing the physical characteristics of the metal surface somewhat. Abrasion, even by a sharp wire brush undoubtedly work hardens the surface to some extent.

There is considerable interest in the effect of proton bombardment since the proton flux in space and on the moon is apparently sufficient to remove surface oxides at least from some metals in reasonable periods of time. In fact, proton bombardment is the only mechanism aside from abrasion by which most metals could lose their oxide films in reasonable periods of time in space except at high temperatures. Therefore, although proton bombardment is not included in the present plans it is hoped that the ion guns will prove suitable for operation with hydrogen as well as with xenon, and for operation at the 10 kv or so required for proton flux space simulation. The flux density of protons in space is low enough to be easily exceeded in the laboratory.

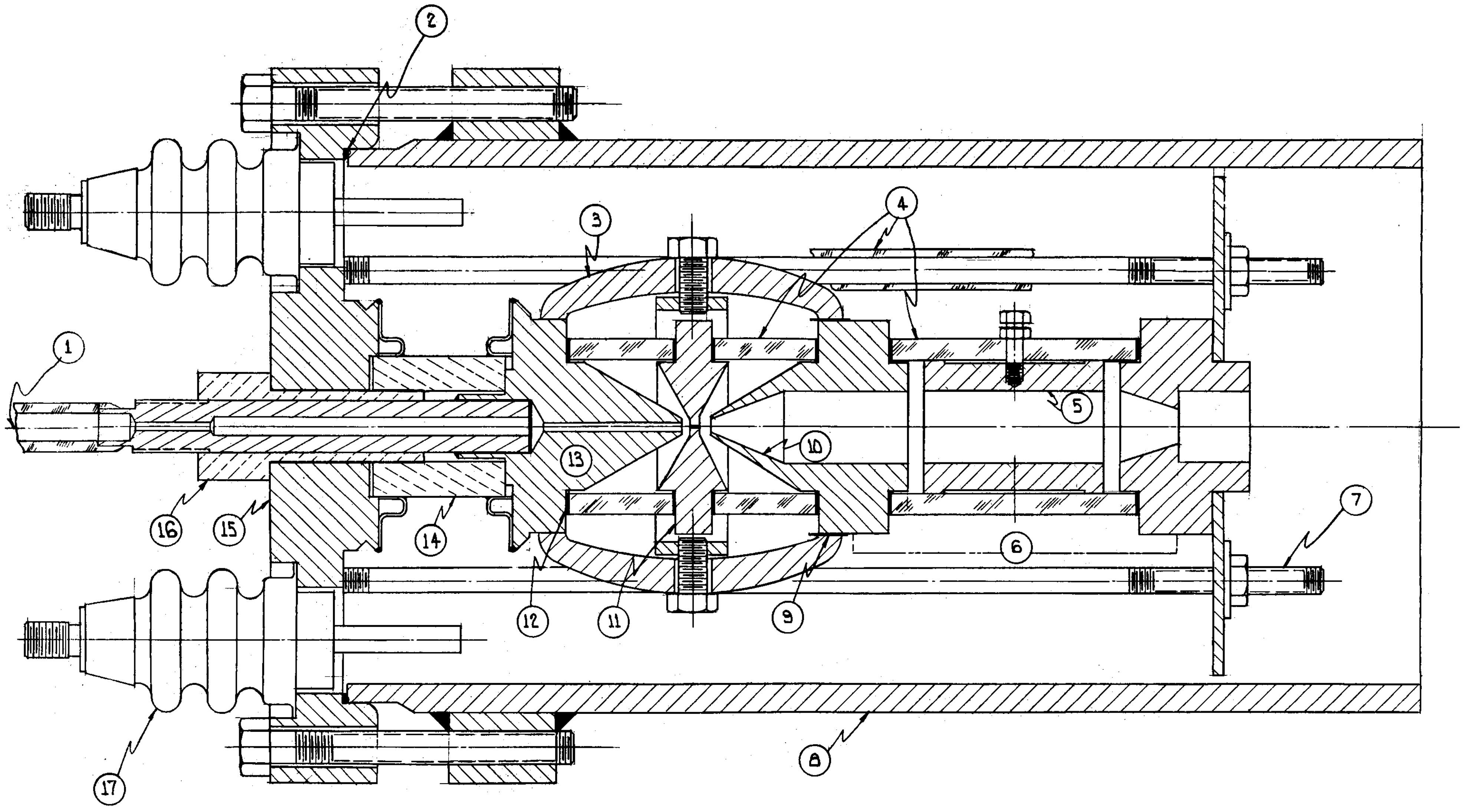
ION GUN DESIGN

Design of the ion guns was preceded by considerable study of the literature. None of the commercially available units were suitable for direct attachment to the apparatus. Therefore, a special designed gun is being fabricated. The design is shown schematically in Fig. 3.

Pure xenon gas will be bled in at the rate required to maintain a pressure of 10^{-2} torr to 10^{-1} torr, on the inlet side of the 0.010-inch diameter orifice in the aluminum orifice plate. A glow discharge will be established by applying a suitable voltage between the orifice plate and the conical pole piece through which the gas enters. An intense magnetic field is established between this conical pole piece and the conical pole piece on the other side of the aluminum orifice plate, by means of the permanent magnets clamped to the hexagonal forces of these pole pieces, which are composed of an iron-cobalt alloy with high saturation flux density. Mica will be placed under one end of the magnets for electrical insulation. The second pole piece also serves as the ion extractor which may require as much as 30 kv for efficient extraction. These ions then pass through an Einzel lens with its center section in three segments to permit beam deflection as well as focussing.

LEGEND FOR FIGURE 3 - ION GUN

1 Gas Inlet
2 Gold "O" Ring
3 Magnets (six)
4 Glass Tubes
5 Aluminum Deflectors (Three Sectors)
6 Einzel Lens
7 Tie Rods
8 Stainless Steel Pipe (4" diameter)
9 Mica
10 Ion Extractor (and pole piece)
11 Aluminum Orifice Plate
12 Aluminum Foil
13 Pole Piece (Fe-Co) (Hexagonal Faces)
14 Ceramic Insulator
15 Stainless Steel Plate
16 Boron Nitride Bushing
17 Ceramic Insulators (six)



ION GUN

The whole assembly including the magnets (which are polished) is located within the vacuum system, the discharge chamber being isolated by aluminum gaskets between the flat ground ends of the tubular glass insulators and machined faces on the pole piece and orifice plate. The assembly is mounted on a thick stainless steel plate which fits on the end of the 4-inch diameter vacuum chamber side arm. A gold "O" ring .030-inch in diameter will be used for the vacuum seal at this point. All electrical connections are brought out through ceramic-to-metal sealed insulators, mounted in the plate. Construction is such as to permit baking out at temperatures usually used for Pyrex glass.

SPECIMEN PREPARATION

The specimen design is shown in Fig. 4. Eight such specimens will be mounted on each of the indexing wheels shown in Fig. 2. The specimens meet each other with their axes at right angles to form a cross. The face width will be such as to permit the softer specimen to yield at less than the 2000 lbs. of force available, but the width of the two specimens in a given pair will be equal, i.e. it will be determined by the compressive yield strength (hardness) of the softer piece. Before they are beveled the specimen faces will be finished on 000 metallographic polishing paper on a flat glass plate rubbing in one direction and using a jig if necessary to insure flat accurately oriented faces. They will then be beveled to the desired face width by machining or surface grinding. Finally, they

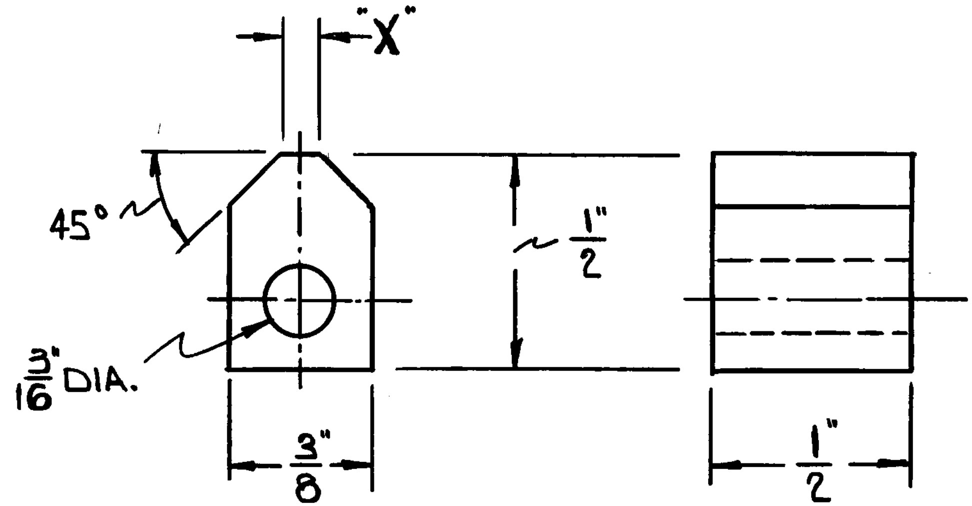


FIGURE 4 - Adhesion Specimen

will be carefully cleaned and degreased. Before they are joined they will, of course, have been baked out in vacuum along with the rest of the vacuum system at 150 to 300°C and will finally have been either wire brushed or ion bombarded.

The hard specimens of O.F.H.C. copper and 1018 steel will be made directly from cold drawn 3/4-inch diameter bar stock now on hand. The soft copper and soft 1018 steel specimens will be made from the same stock after annealing. The 3/4-inch diameter titanium stock could be obtained only in the annealed condition. A portion will be cold forged to harden it so that both the hard and the soft conditions will be represented. The Cu-Be alloy (No. 25) will be machined to size from 3/4-inch diameter bar stock in the solution treated condition and some of the specimens hardened by the standard aging treatment prior to finishing on the 000 paper and beveling by grinding. The 440C stainless steel and the 4140 steel specimen will be machined to size from soft bar stock except for .010 inches to be removed from the face of the hard ones by surface grinding to remove decarburized metal prior to finishing on the 000 paper. The 4140 steel will be drawn to about 50 Rockwell C. The 440C steel and Cu-Be will be left as hard as possible.

AUXILIARY EQUIPMENT

The power supplies for the ion guns remain to be selected, located and reserved, purchased or rented. The specific requirements are still being assessed. Both high and low voltage supplies will be required but no high power supplies should be necessary.

Gas metering devices are also required for the ion guns. Some but not all of the necessary components are on hand.

The load cell and companion recording equipment and the optical projection equipment is on hand.

EXPERIMENTAL PROCEDURES

Four soft copper specimens and four soft 1018 steel specimens will be loaded into each wheel for the copper to copper and steel to steel tests using wire-brush cleaning. This test can proceed before installation of the ion guns and their auxiliary equipment.

With the system pumped down and baked out, and the specimens about 3/16 inches apart, a 1/4 inches wire brush will be pushed back and forth between them a few times. They will then be pushed together till they deform slightly as shown by the optically projected 20X silhouette. They will then be pulled apart and the cohesion force measured.

The apparatus will then be reloaded in the same way and the experiment repeated using ion bombardment cleaning. (Considerable experimentation with the ion guns may be necessary between these two runs.) The specimens facing the guns will be bombarded, indexed to position to face each other, then pushed together and pulled apart as before. The xenon flow will be stopped immediately on completion of the cleaning operation. It is anticipated that the total pressure in the system will drop at least a decade between cleaning and joining. The specimens may be cleaned in quick succession rather than simultaneously to simplify the operation and equipment.

Whether the ion bombardment or wire-brushing will be used in the remainder of the tests will depend on the results of these two runs and such additional confirming runs as may be necessary.

Task D, E and F as set forth in the introduction will then be undertaken.